(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Gerhard Meyer et al.

Application Ser. No. 10/539,877 — Confirmation No.: 6327

Filed: December 8, 2003 — Art Unit: 1794

For: Fire Protection Means and Method for the Production Thereof — Examiner: Elizabeth A. Robinson

DECLARATION UNDER 37 C.F.R. §1.131

Valentino Villari, hereby states as follows:

1. I am an inventor of the invention described and claimed in the above-identified application, and am personally familiar with the documents attached hereto and the facts recited herein.

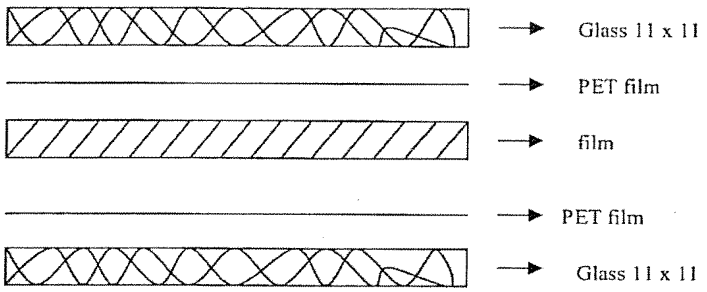
2. Attached as an exhibit are copies of documents describing and illustrating preparation of the invention disclosed and claimed in the above-identified application.

3. The dates on the exhibit have been deleted, but each is prior to September 18, 2002.

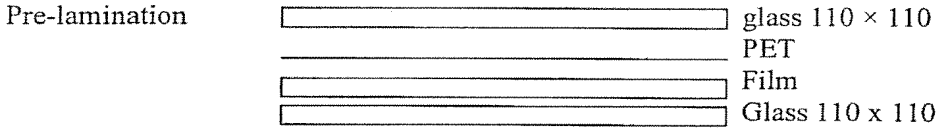
4. As shown in the exhibit, a fire protection means and fire protection glazing falling within the scope of the invention were prepared before September 18, 2002.

5. More specifically, the fire protection means comprised a hybrid film system, consisting of two or three layers. The two- and three-layered hybrid film system consisted of a film made from a mixture of waterglass and glycerin as intumescent material. This film was covered on one or both flat sides with PET-films (polyethylene-terephthalate) to yield a hybrid film system.

The two- and three-layers comprising hybrid film systems were put in between two sheets of glass, fixed with silicone glue and laminated. As shown in the laboratory notes, the glazing with three-layer hybrid film system had the following composition/build-up:



The fire glazing with a two-layer comprising hybrid system looked as follows:



In addition to the two different layer built-ups of the hybrid film system, the composition of the glycerine/waterglass film used in both cases as organic/ inorganic mixture was varied as shown in the following table to examine its impact on overall glazing properties, especially with respect to haze and transparency:

	WG	Glycerin calculated	solid material	added	%
1%	127.59	1.289	37.07	1.28	1.00
3%	127.82	3.952	38.34	3.94	3.00
7%	124.89	9.400	40.88	~~9.39~~ 9.40	7.00
9%	111.57	11.034	42.15	11.11	9.00

Summarizing the above, fire protection glazings were prepared, consisting of two- or three-layered hybrid film systems laminated between two glass sheets. The hybrid film systems used consisted of an intumescent layer made from glycerine and waterglass in different ratios, i.e. a mixture of an organic and an inorganic component. The second and respectively third film of the hybrid film systems were made from PET, i.e. an organic polyester material, so that the intumenscent layer

and the PET-layer differed from each other with respect to their components/composition. The glazings prepared were transparent in the visible spectrum. Accordingly, the invention was already made at that time.

6. The acts described above in paragraph 4 and 5 were carried out in the Netherlands.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made punishable by fine or imprisonment, under Section 1001 of Title 18 of the United State Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

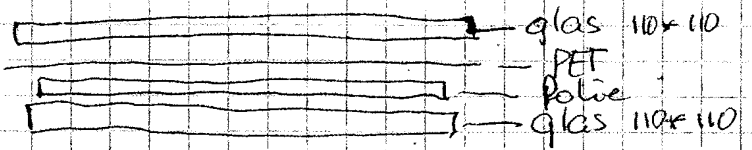
01.02.10
Date

Name: Valentino Villari

EXHIBIT

[redacted] uitnieuwen.

voorlaminieren.



1 × met platgewalste folie (zie 4-10-a). [redacted] P-A

1 × met "verse folie" van substraat. [redacted] P-B.

A 2 × glas + 1 × PET + folie [redacted] P-A. 242,53g.

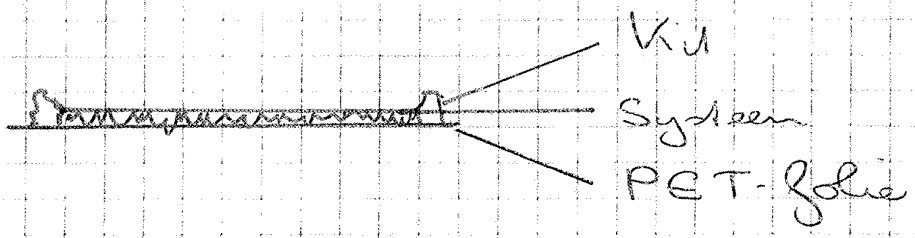
B " " " [redacted] P-B 242,22g.

11.10 in voorverwarmde stoof bij 80°C met 2 × voorverwarmde 4 metalen platen erop weggezet.

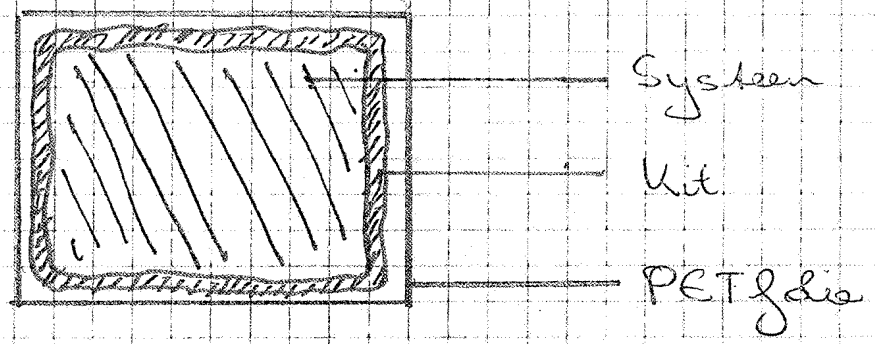
4G met glycerine 1, 3, 7, 9 %.

	WG	glycerine berekend	vaste stof toegevoegd.		%
1%	127.59	1,289	37.07	1.28	1.00
3%	127,82.	3.952	38.34	3,94.	3.00
7%	124,89.	9,400	40.88	~~9,39~~ 9,40	7.00
9%	111,57.	11,034.	42,15	11.11.	9.00

Zijaanzicht opbouw substraat



bovenaanzicht opbouw substraat



Berekeningen

In de onderstaande tabel staan de resultaten van de berekeningen herhaald.

Staal	Substr.	130nm	205nm	230nm	265nm	315nm	340nm
A	glas	50,7	37,10	35,6		30,8	29,7
B	glas	46,8	33,8	32,7	39,8	29,1	-
C	glas	44,3	30,3	29,4	28,0	-	-
d	PET	-	-	29,7	28,3	-	-
E	PET	-	-	31,6	-	-	27,5
f	PET	-	-	33,7	-	-	27,9

Restpercentages voor droger.

NR	% Na_2O	% SiO_2	% H_2O	% glij	% Zr-Compl. 1	% Zr-Compl 2
A+d	7,7	25,4	61,8	5,1	-	-
B+E	7,8	24,5	62,6	5,0	0,7	-
C+f	8,0	24,4	62,4	4,9	-	0,26

Na Complex oxidatie.

NR	% Na_2O	% SiO_2	% ZrO_2
A+d	23,3%	76,7%	-
B+E	24,1%	75,3%	0,53%
C+f	24,6%	74,6%	0,80%

[redacted] Folie bereiding 100 + 5% glycerine ter verontreiniging.

VS% 39,61%	I	II	III	IV	V	VI
opp substraat	99,50	100,80	99,00	98,57	72,91	72,93
massa substraat	124,7661	125,3161	125,9719	123,8530	46,5795	46,6204
inweeg bereikend	20,72	20,93	20,62	20,52	15,19	15,20
inweeg.	20,72.	20,95	20,67.	20,54.	15,24.	15,22.
massa bij 25%	135,71	136,38	136,89	134,70	54,47	54,23

Dikte + Haze [redacted] G

V zonder	met	film	T	H	zonder	met	film	T	H
1,58	2,07	0,51	91,8	0,11	1,56	2,04	0,48	91,6	0,28
1,58	2,07	0,51	91,4	0,20	1,57	2,03	0,46	91,7	0,18
1,58	2,14	0,58	91,9	0,18	1,56	2,09	0,53	91,9	0,13
1,57	2,15	0,58	91,7	0,17.	1,58	2,18	0,60	91,7	0,34.

9.50 O_2 100 l/h. ~~oven aan~~.

10.10 oven aan.

10.35 oven 80 °C.

16.35 (360)	I	II	III	IV	V	VI
	135,95	136,03.	137,34.	135,16.	55,00	54,99.
17.35 (420)	135,77.	136,52	137,08	134,88	54,80	54,77.
18.35 (480)	135,66	136,41	136,90	134,72	54,68	54,66.

laminieren [redacted] P-B

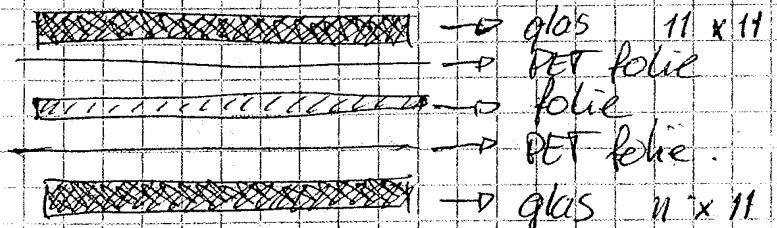
glasplaten 100 · 100 voorwarmen op 80°C

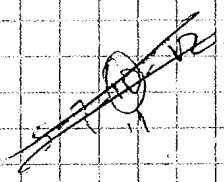
folie 15 minuten in PE ingesealed voorwarmen bij 80°C

folie tussen de 2 warme glasplaten. 4 klemmen erop en

terug in de stoof →  !

experiment: Folie plat maken voor laminieren.





folie [redacted] PP-C.

totaalgewicht 245.75 g

drukgewicht: 3003,18 g

3029,83 g

foto's voor [redacted] PP-C01

[redacted] PP-C02.

13.30 in stoof bij 80°C.

16.00 gewicht 245,71 g.

foto's.

17.30

Haze WG3		Haze WG4	
Haze	Trans	Haze	Trans
5,77	85,6	5,94	87,3
23,7	84,6	5,58	87,5
4,29	85,8	1,17	87,9

~~04~~ foto's na [redacted] PP-C03

[redacted] PP-C04

in A4 map onder glas weggelegd.

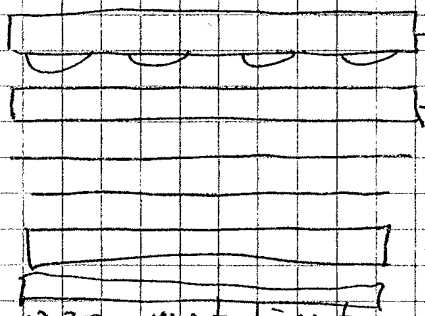
g	massa		% vocht	
massa tijd	WG 1	WG 2	WG 1	WG 2
9.10	118.87	118.76	36.8	21.8
11.10	118.43	118.74	32.9	21.5
13.50	118.00	118.71	28.7	21.1

puntbelasting [redacted]

t	W	A	B	C	D
0	206,5	0	0		0
1	+100	2,49	1,78		2,51
2	"	4,30	2,36		3,05
3	"		2,14 scheurt		2,20 scheurt
4	"				

A begint meteen te scheuren.
C ook

kanmineren [redacted] P-FC.



12.30 met lijmtangen bij 80°C in stoof gezet.

13.45 PET vervangen door 100 x 100 glasplaat.

kGn vlf 124 ~~106,15~~ 24,60

1,6-BDO ~~5,50~~. (1,29) 0,46 + 0,84g na oplossen ± 3,5 u

demi 5,59

~~2,99~~ 1,29
~~11,60~~ 0,46
0,83

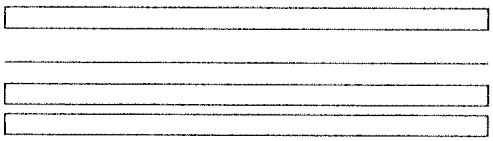
vaste stofgehalte 32,59

1,4-BDO : 4,1283 %

ENGLISH TRANSLATION OF EXHIBIT

Lamination

Pre-lamination



1 × with flattened film (see [redacted]) [redacted] P-A

1 × with "fresh film" of substrate 011001P-B

A 2 × glass + 1 + PET + film [redacted] P-A 242.53 g

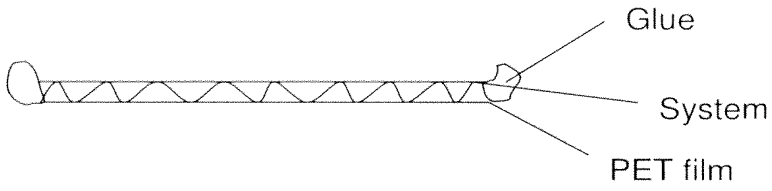
B " " " " " [redacted] P-B 242.22 g

No. 10 in pre-heated kiln at 80 °C [176 °F] put away with 2 x pre-heated metal plates on it

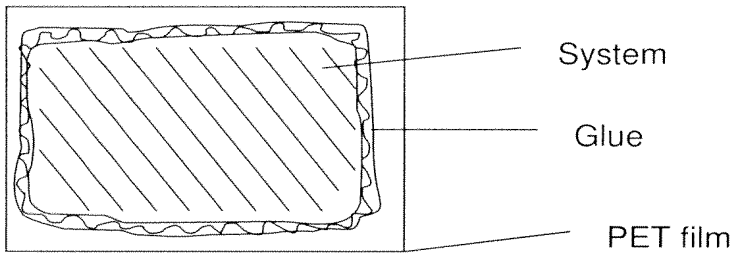
WG (waterglass) with glycerin 1%, 3%, 7%, 9%

	WG	glycerin calculated	solid material	added	%
1%	127.59	1.289	37.07	1.28	1.00
3%	127.82	3.952	38.34	3.94	3.00
7%	124.89	9.400	40.88	~~9.39~~ 9.40	7.00
9%	111.57	11.034	42.15	11.11	9.00

Side view of substrate structure



Top view of substrate structure



Calculations

The table below shows the results of the core moisture calculations

Dish	Substrate	130 m	205 m	230 m	265 m	315 m	340 m
A	glass	50.7	37.10	35.6		30.8	29.7
B	glass	46.8	33.8	32.7	39.8	29.1	-
C	glass	44.3	30.3	29.4	28.0	-	-
D	PET	-	-	29.7	28.3	-	-
E	PET	-	-	31.6	-	-	27.5
F	PET	-	-	33.7	-	-	27.9

Remaining percentages before drying

No.	% Na_2O	% SiO_2	% H_2O	% glyc.	% Zr Compl. 1	% Zr Compl. 2
A + D	7.7	25.4	61.8	5.1	-	-
B + E	7.8	24.5	62.6	5.0	0.17	-
C + F	8.0	24.4	62.4	4.9	-	0.26

After complete oxidation

No.	% Na_2O	% SiO_2	% ZrO_2
A + D	23.3%	76.7%	-
B + E	24.1%	75.3%	0.53%
C + F	24.6%	74.6%	0.80%

Film preparation WG + 5% glycerin for purposes of point loading

VS% 39.61%	I	II	III	IV	V	VI
substrate surface	99.50	100.50	99.00	98.51	72.91	72.99
substrate weight	124.7661	125.3161	125.9719	123.8530	46.5795	46.6204
calculated weighed-in quantity	20.72	20.93	20.62	20.52	15.19	15.20
weighed-in quantity	20.72	20.95	20.67	20.54	15.24	15.22
weight at 25%	135.71	136.38	136.89	134.70	54.47	54.23

Thickness + haze [redacted] G
V

without	with	film	T	H	without	with	film	T	H
1.56	2.07	0.51	91.8	0.11	1.56	2.04	0.48	91.6	0.28
1.56	2.07	0.51	91.4	0.20	1.57	2.03	0.46	91.7	0.18
1.56	2.14	0.58	91.9	0.18	1.56	2.09	0.53	91.9	0.13
1.57	2.15	0.58	91.7	0.17	1.58	2.18	0.60	91.7	0.34

9:50 a.m. O_2 400 l/h ~~oven on~~
10:10 a.m. oven on
10:35 a.m. oven 80 °C [176 °F]

	I	II	III	IV	V	VI
4:35 p.m. (360)	135.95	136.69	137.34	135.16	55.00	54.99
5:35 p.m. (420)	135.76	136.52	137.08	134.88	54.80	54.77
6:35 p.m. (480)	135.66	136.41	136.90	134.72	54.68	54.66

Lamination [redacted] P-B

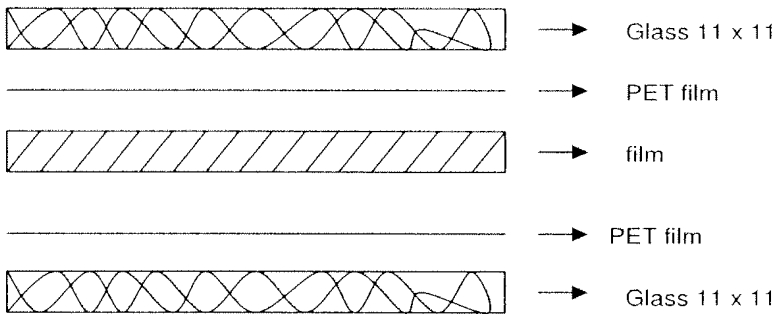
Glass plates 100 × 100 pre-heated to 80 °C [176 °F]

Film sealed in PE for 15 minutes, pre-heated to 80 °C [176 °F]

Film between the two hot glass plates, 4 clamps on them and back into the kiln

⇨ 👍 !

Experiment: flattening the film prior to lamination



Film [redacted] PP-C
total weight: 245.75 g
pressure weight: 3003.18 g
3029.83 g
Photos for [redacted] [redacted] PP-C01
[redacted] PP-C02

1:30 p.m. into the kiln at 80°C [176°F]

4:00 p.m. weight 245.71 g
photos

5:30 p.m.

Haze	WG3		WG4	
	Haze	Trans.	Haze	Trans.
	5.77	85.6	5.94	87.3
	23.7	84.6	5.58	87.5
	4.29	85.8	1.17	87.9

~~Ok~~ photos after [redacted] PP-C03
[redacted] PP-C04

Filed in A4 folder under "glass"

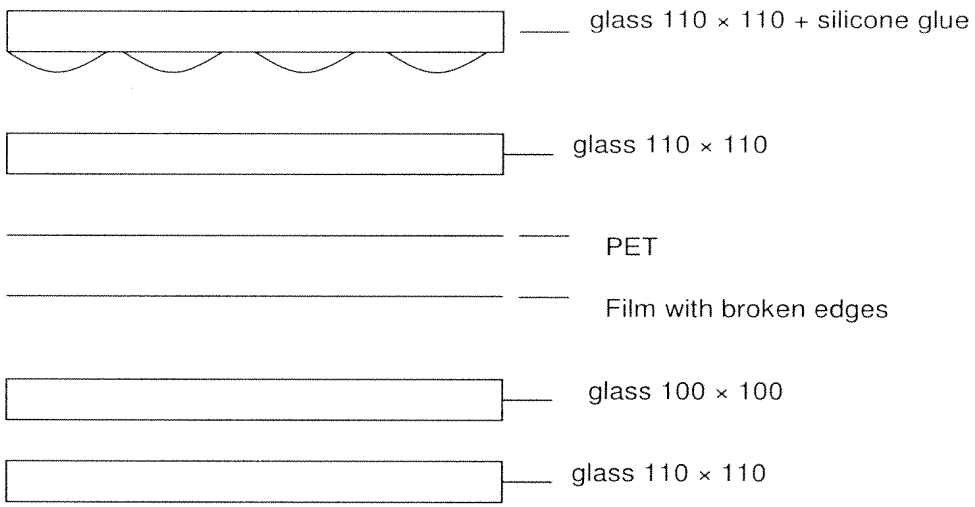
		Weight		% Moisture	
~~weight~~	Time	WG1	WG2	WG1	WG2
	9:10 a.m.	118.87	118.76	36.8	21.8
	11:10 a.m.	118.43	118.74	32.9	21.5
	13:50 a.m.	118.00	118.71	28.7	21.1

Point loading [redacted]

T (time)	M (mass)	A⇧	B⇧	C⇩	D⇩
0	206.5	0	0		0
1	+ 100	2.49	1.78		2.51
2	"	4.30	2.36		3.05
3	"	2.14 tear			2.20 tear
4	"				

A begins to tear immediately
C as well

Lamination [redacted] P-C



12:30 p.m. placed into the kiln at 80 °C [176 °F] with clamping tool

1:45 p.m. PET replaced with 100 × 100 glass plate

WG ref. 124 ~~106.12~~ 24.60
1.6-BDO ~~(5,58)~~ (1.29) 0.46 + 0.84 g after dissolving ± 3.5 hrs.
demi (demi-water) 5.59

[*illegible*]

~~1.59~~
~~0.60~~
~~0.88~~

Solid material content 32.59
1.4-BDO 4.1283%